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# THE UNIVERSITY OF MICHIGAN

COLLEGE OF ENGINEERING  
DEPARTMENT OF CHEMICAL AND METALLURGICAL ENGINEERING

## **Screening Program on Superalloys for Trisonic Transport**

Report No. 2

## **Results for Cold-Worked N155 Alloy**

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Prepared for:

National Aeronautics and Space Administration  
Grant NsG-124-61  
Washington 25, D. C.



Administered through:

November 15, 1961

OFFICE OF RESEARCH ADMINISTRATION • ANN ARBOR

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SCREENING PROGRAM  
ON SUPERALLOYS FOR TRISONIC TRANSPORT

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ORA Project 04368



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RESULTS FOR COLD-WORKED N155 ALLOY

SUMMARY

N155 alloy sheet cold reduced 40 and 65 percent was subjected to a screening program in the as-rolled condition designed to rate materials for possible usefulness for the airframe of a trisonic transport plane.

Cold reductions of 40 to 65 percent produced the following properties at room temperature in the as-rolled N155 sheet investigated:

	<u>40-percent reduction</u>		<u>65-percent reduction</u>	
	<u>L*</u>	<u>T</u>	<u>L</u>	<u>T</u>
Tensile strength (psi)	188,000	190,000	217,000	232,000
0.2% offset yield strength (psi)	167,000	159,000	185,000	187,000
Elongation (%)	7	8	4	5
Sharp notch tensile strength (psi)	183,000	168,000	186,000	115,000
N/S ratio	.97	.88	.86	.50

\* L - longitudinal, T - transverse

The strength and ductility of unnotched samples decreased from a maximum at -110°F to a plateau from 350° to 800°F. The strength of samples with sharp-edge notches decreased more at the higher temperatures so that the N/S ratio decreased. Other than the higher strength and lower ductility of the material reduced 65 percent, the

main difference from the material reduced 40 percent was the lower strength and N/S ratio of transverse specimens in comparison to longitudinal specimens.

Exposure for 1000 hours at 650°F under 40,000 psi caused an increase in strength at room temperature. The properties were not changed at 650°F. Exposure also resulted in a measurable decrease in length of unnotched samples.

The data presented are very limited and are intended only for comparison with data taken for other alloys being similarly evaluated.

## INTRODUCTION

The evaluation of sheet materials for construction of the airframe of a trisonic transport plane is the subject of an experimental program being conducted cooperatively by several laboratories. At the University of Michigan, the possible utility of heat-resistant alloys, known as "superalloys", in sheet form is being studied.

The airframe of the aircraft would be subject to aerodynamic heating during a prolonged service life. In addition, there would be the usual stressing at ambient temperatures during take-off and landing. A suitable material for such an application must possess adequate original strength and must be sufficiently stable under dynamic heating during service to maintain adequate properties. It is important that the material possess and maintain an acceptable resistance to rapid crack growth.

The first phase of the investigation is to screen a number of promising alloys. Test conditions are limited to the very minimum which will permit preliminary comparison of the materials. Tensile tests are being conducted at  $-110^{\circ}$ ,  $75^{\circ}$ ,  $350^{\circ}$ ,  $650^{\circ}$ , and  $800^{\circ}\text{F}$  on unnotched specimens and specimens with sharp-edge notches. Retention of original properties is evaluated by exposing specimens for 1000 hours at  $650^{\circ}\text{F}$  under 40,000 psi tensile load and then repeating the tensile tests at  $75^{\circ}$  and  $650^{\circ}\text{F}$  with both unnotched and notched specimens. Creep measurements are made during exposure of unnotched specimens.

The choice of these test and exposure conditions has been extremely arbitrary. The data obtained are only expected to delineate the characteristics of the material for screening purposes. Actual service conditions are not well enough established at this time to permit formulation of a direct relation between service requirements and the results of the screening program.

This report is the second in a series (ref. 1) and presents data for cold-worked N155 alloy in the form of 0.025-inch thick sheet. No heat treatment was applied to the material prior to testing. A relatively large degree of cold work (up to 65 percent) was applied to develop tensile and yield strengths of interest for the application. N155 is an alloy which has tensile and yield strengths which are too low for the application unless it is cold worked.

The investigation is being carried out under the sponsorship and with the financial assistance of the National Aeronautics and Space Administration. It is being coordinated with similar programs of the NASA and other interested groups in the same field. The test conditions of the screening program are based on NASA experience in this area.

## EXPERIMENTAL MATERIAL

The N155 alloy was made by the Wallingford Steel Company as 0.025 inch sheet in three conditions of cold reduction: 40 percent, 55 percent, and 65 percent. The material was from Heat M-5623 with the following reported chemical composition (weight percent):

<u>C</u>	<u>Mn</u>	<u>P</u>	<u>S</u>	<u>Si</u>	<u>Fe</u>	<u>Cr</u>	<u>Ni</u>	<u>Co</u>	<u>Mo</u>	<u>W</u>	<u>Cb+Ta</u>
.11	1.61	.017	.011	.72	Bal	22.14	19.91	19.50	3.22	2.40	1.21

The rolling was done on a strip mill providing material 12 inches wide. Reported reduction histories were as follows:

<u>Reduction (percent)</u>	<u>Initial Thickness (inch)</u>	<u>Number of Passes</u>
40	0.041	3
55	0.054	5
65	0.072	11

All testing was done on specimens machined from the as-rolled stock without any heat treatment.

Representative photomicrographs are shown in figure 1 for the alloy with 40 percent and 65 percent cold reduction. In order to delineate the grain boundaries in these materials, an age at 1200°F for five hours was necessary. The grain structure revealed by this aging treatment should be essentially the same as that in the as-rolled condition. The distribution of grain sizes was quite uniform through the thickness of the sheet and is well represented by the micrographs of figure 1.

## EXPERIMENTAL PROCEDURES

The alloy was evaluated using three basic criteria:

- (1) The level of tensile properties obtainable in the alloy as a function of test temperature, using temperatures of  $-110^{\circ}$ ,  $75^{\circ}$ ,  $350^{\circ}$ ,  $650^{\circ}$ , and  $800^{\circ}\text{F}$ .
- (2) Resistance of the material to catastrophic crack growth at the same temperatures using a specimen with sharp-edge notches.
- (3) Maintenance of mechanical properties after exposure under stress at elevated temperature. The exposure used was 1000 hours at  $650^{\circ}\text{F}$  under 40,000 psi. Subsequent tensile tests were conducted at  $75^{\circ}$  and  $650^{\circ}\text{F}$ .

Properties in both the longitudinal and transverse directions were measured to avoid misleading results from anisotropy effects which might be present.

### Test Specimens

All test samples were prepared from the as-rolled stock.

#### Unnotched Specimens

The configuration of the specimens used to measure unnotched properties is given in figure 2a. The reduced section was 0.5 inch wide by 2.0 inches long. Specimens were prepared from rectangular blanks by milling. Ten specimens were machined at a time, using a fixture to clamp the blanks together and assure accurate alignment throughout the machining operations.

#### Notched Specimens

Resistance of the material to catastrophic crack growth was evaluated using the specimen with sharp-edge notches recommended



by the ASTM (ref. 2). The configuration of this specimen is shown in figure 2b. As with the unnotched specimens, ten blanks were machined at one time, using a second fixture to maintain alignment. The 1-inch reduced section was first milled to size. The notches were then ground almost to size with an alundum wheel dressed to a sharp point on a 60-degree included angle. The notch root was then finished by manually drawing a sharp carbide tool through the notches, using a shaper, until the required depth was achieved. The root radii and net-section width were then measured using a 50x optical comparator. Root radii varied from 0.0005 to 0.0007 inch.

The specimens in figure 2 are shown with two pinhole sizes. The larger size was used for the early work. Occasional failure of the specimens at the pinhole was then encountered, necessitating a change to the smaller diameter pins.

### Test Procedures

The test procedures followed those of references 2 and 3.

#### Tensile Tests

All tensile testing was conducted with a 60,000-pound capacity hydraulic tensile machine. Unnotched samples were strained at an approximate strain rate of 0.01 inch per inch per minute, up to about 2 percent deformation. The strain rate was then increased to about 0.05 inch per inch per minute, until failure. Notched samples were loaded at a rate of 1000 psi net section stress per second.

Tests at -110°F were run in a mixture of dry ice and acetone. Temperature was checked with a thermometer for all tests. The specimens for tests at elevated temperature were heated with an electric resistance furnace. Temperature variation along the gage length, as measured by a thermocouple at each end and at the center,

was held to within  $\pm 5^{\circ}\text{F}$ . Indicated test temperature was within  $\pm 3^{\circ}\text{F}$  of the nominal temperature for all tests.

Strain measurements were made on all unnotched samples. At room temperature, a microformer-type stress-strain recorder with a sensitivity of 0.0001 inch per inch was employed. At all other temperatures, strain measurements were made with a modified Martens-type optical-lever extensometer. The extensometer was attached by collars clamped onto the gage section. For the specimens being tested, this system has a sensitivity of about 0.000005 inch per inch.

#### Exposure Tests

The exposures were conducted in air on both unnotched and notched specimens using conventional, beam-loaded creep-rupture machines. Temperature was maintained with an electric resistance furnace to the same limits as described above for the tensile tests at elevated temperature. Strain measurements throughout the exposure period were made on all unnotched specimens using the extensometer system described above. For the specimens being tested, this system has a sensitivity of about 0.000005 inch per inch. After exposure, specimens were tensile tested without any further machining or surface treatment.

## RESULTS

The tensile properties of unnotched samples were evaluated at 75° and 650°F for the alloy with all three amounts of cold work, 40, 55, and 65 percent. The results indicated that the complete screening program could be confined to materials cold worked 40 and 65 percent. No heat treatments were applied prior to testing.

### Tensile Properties as a Function of Amount of Cold Work

Cold reductions of 40 and 65 percent produced material with the following properties (table I) in the longitudinal direction at room temperature:

Cold reduction (percent)	40	65
Ultimate strength (psi)	188,000	217,000
0.2% offset yield strength (psi)	167,000	185,000
Elongation (percent)	7	4
Notched tensile strength (psi)	183,000	186,000
N/S ratio	0.95	0.86

The strength of unnotched specimens at room temperature increased uniformly (table I and figure 3) with amount of reduction. Elongation decreased from 7 to 4 percent. The strength of notched specimens did not change appreciably with the result that the N/S ratio decreased.

The properties at 650°F (table I and figure 3) followed the same general pattern as they did at room temperature with the following differences:

- (1) Strengths were at lower levels.
- (2) The strength of notched samples was lower in relation to that of unnotched samples with the result that the N/S ratio was lower than it was at room temperature.

(3) There was a slight increase in N/S ratio at 650°F with increasing cold reduction from 0.65 for material with 40 percent to 0.73 for a reduction of 65 percent.

(4) Elongation was only 1.5 to 2 percent.

### Screening Tests

Since the properties changed quite uniformly with increasing cold reduction, the screening program was limited to the materials cold reduced 40 and 65 percent.

The material with 40 percent cold work gave the following results (table I and figures 4 and 5) for screening tests:

(1) The strength and ductility of unnotched specimens was considerably higher at -110°F than at room temperature. Tensile strength of notched specimens was also higher but the N/S ratio was about the same.

(2) The decrease in strength and ductility of unnotched specimens with increasing test temperature, practically all occurred between -110° and 350°F. There was little change from 350° to 800°F.

(3) Notched tensile strength decreased with increasing temperature over the entire temperature range such that the N/S ratio also decreased as the temperature was raised.

(4) Exposure at 650°F for 1000 hours under 40,000 psi did not change properties appreciably. Strength at room temperature was slightly higher after exposure while ductility was slightly reduced. Properties at 650°F were not affected.

(5) There was little difference between longitudinal and transverse specimens except that the strength of notched samples and the N/S ratios were slightly lower for transverse specimens.

The screening tests on material cold reduced 65 percent gave results (table I and figures 6 and 7) which were qualitatively similar

to those for a reduction of 40 percent but at the different levels resulting from the higher cold reduction. The only other major difference between the results for the two materials was the greater anisotropy shown in the material cold reduced 65 percent. This effect was most evident in the notched tensile strength and N/S ratio which were much lower in the transverse direction than in the longitudinal direction.

#### Dimensional Stability During Exposure at 650°F

Creep measurements during exposure of unnotched specimens at 650°F for 1000 hours under 40,000 psi showed no positive creep (figure 8). The longitudinal specimens of material reduced 40 percent underwent no significant change in length. The transverse specimens gave results which suggested a decrease in length of about 0.01 percent, most of which occurred during the first 100 hours. The material reduced 65 percent gave strain measurements which indicated about 0.01-percent decrease in length for longitudinal specimens and 0.02 percent for transverse specimens. Most of this change occurred during the first 100 hours for the longitudinal samples. About half of the change occurred during the first 100 hours in the transverse samples.

It is doubtful that the alloy is subject to positive creep at 650°F under 40,000 psi. The measured decreases apparently were due to volume decrease which was more in the transverse than in the longitudinal direction.

## DISCUSSION

Although data were not taken in the present investigation for material without cold work, the tensile properties of N155 alloy were considerably enhanced by the cold reduction. Comparison of the properties as reported in this study with those for annealed sheet (ref. 4) at room temperature shows that the ultimate tensile strength was approximately doubled and the 0.2 percent offset yield strength was nearly tripled by 65 percent cold work. The elongation was decreased considerably from about 40 percent for annealed sheet to the 4 to 7 percent level for the sheet with cold work.

The cold working procedures used resulted in the properties of transverse notched specimens being significantly lower than those of longitudinal notched specimens in the material reduced 65 percent. The effect was not large in the material reduced 40 percent.

The properties fell off with increasing temperature from  $-110^{\circ}$  to  $350^{\circ}\text{F}$  with little further change to  $800^{\circ}\text{F}$ . The general improvement in properties as test temperature is decreased to  $-110^{\circ}\text{F}$  seems characteristic of superalloys.

There was no deterioration of properties at room temperature or  $650^{\circ}\text{F}$  from exposure at  $650^{\circ}\text{F}$  for 1000 hours under 40,000 psi. The strength of unnotched specimens at room temperature increased with a slight decrease in elongation. There was no change at  $650^{\circ}\text{F}$ . Except for the slight decrease in elongation, properties were either unchanged or improved.

Two features of the results are shown by figure 9 which presents the relation at room temperature between N/S ratio and the tensile properties from unnotched samples for the material before and after exposure at  $650^{\circ}\text{F}$ :

- 1) As strength was increased by cold work, an associated

drop in N/S ratio resulted.

2) Comparison of the curves for as-rolled and exposed material shows that the main effect of exposure was to shift the curves to higher strength levels without changing the N/S ratio a great deal. This suggests that a low temperature heat treatment of the material might produce a better combination of strength and N/S ratio than was obtained by cold work alone.

As previously discussed, exposure did not cause a similar change in level of strength at 650°F. There was, however, a decrease in N/S ratio as the strength level increased from cold work.

The microstructure of samples with both 40 and 65 percent cold work was checked after exposure at 650°F. As was the case for the as-rolled material, etching failed to produce a resolvable structure in the samples which had been exposed. The samples were then given the aging treatment at 1200°F which was used to delineate the grain boundaries in the as-rolled material. The structures thus produced did not differ in any way from those shown in figure 1 for the as-rolled sheet.

The cold-worked N155 was subject to a slight volume-decrease during exposure at 650°F which resulted in decreased length of the specimens. It is not subject to positive creep at 650°F. Because most of the decrease in length occurred early in the exposure, it is also possible that a low temperature heat treatment might eliminate it.

Recognition should be given to the fact that the data presented in this report are taken from sheet of one thickness (0.025 inch) from one heat. The effects of heat-to-heat and processing variables on the results have not been established.

## CONCLUSIONS

A preliminary evaluation of N155 alloy in the form of 0.025-inch thick sheet with two amounts of cold work was conducted using procedures designed to screen materials for possible use in a trisomic transport plane.

Cold reductions of 40 to 65 percent produced the following properties at room temperature in the as-rolled N155 sheet investigated:

	<u>40-percent reduction</u>		<u>65-percent reduction</u>	
	<u>L*</u>	<u>T</u>	<u>L</u>	<u>T</u>
Tensile strength (psi)	188,000	190,000	217,000	232,000
0.2% offset yield strength (psi)	167,000	159,000	185,000	187,000
Elongation (%)	7	8	4	5
Sharp notch tensile strength (psi)	183,000	168,000	186,000	115,000
N/S ratio	.97	.88	.86	.50

\* L - longitudinal, T- transverse

Strength and ductility decreased from 75° to 350°F with little further decrease from 350° to 800°F. Strength and ductility increased at -110°F.

Increasing the cold reduction from 40 to 65 percent raised strength levels and reduced ductility about the same as from -110° to 800°F. The larger reduction resulted in lower strength of notched transverse specimens than for longitudinal specimens with resultant lower N/S ratios.

Exposure at 650°F for 1000 hours under 40,000 psi did not damage properties. Strength at room temperature was increased. A slight decrease in ductility at room temperature was caused by the exposure. Properties at 650°F were unchanged.



## REFERENCES

1. Rowe, John P., Scoonover, Thomas M., Freeman, James W.: "Results of Screening Tests on Rene' 41 Alloy for Possible Use in the Airframe of a Trisomic Transport". Report No. 1 to National Aeronautics and Space Administration, June 30, 1961, University of Michigan Report No. 4368-1-T.
2. Special ASTM Committee: Fracture Testing of High-Strength Sheet Materials. Chap. I. ASTM Bull. Jan. 1960, pp. 29-40; Chap. II, ASTM Bull. Feb. 1960, pp. 18-28.
3. Manning, C. R., Jr., and Heimerl, G. J.: An Evaluation of Some Current Practices for Short-Time Elevated-Temperature Tensile Tests of Metals. Langley Research Center, Langley Field, Virginia. NASA TN-D-420, September, 1960.
4. "Haynes Alloys for High-Temperature Service", Haynes Stellite Division, Union Carbide and Carbon Division, 1950.

Table I  
TENSILE TEST RESULTS FOR UNNOTCHED AND NOTCHED SPECIMENS

NI55 Alloy - Heat M5623

(Material from 0.025 inch sheet as rolled)

Amount Cold Reduction (percent)	Test Temperature (°F)	Direction <sup>(b)</sup>	Tensile Properties <sup>(a)</sup>									
			As Rolled					After Exposure, 1000 Hrs at 650°F Under 40,000 psi				
			UTS 1000 psi	Y.S. 1000 psi	Elongation percent	N.S. 1000 psi	Notch Ratio	UTS 1000 psi	Y.S. 1000 psi	Elongation percent	N.S. 1000 psi	Notch Ratio
40	-110	L	216	180	12	213	0.99					
		T	222	185	10	194	.87					
	75	L	192	167	6.5	183	.95					
		T	184	--	7.0		.99	205	189	4.5	196	0.96
	350	L	190	159	8.0	168	.88	210	183	6.0	164	.78
		T										
	650	L	173	150	2.5	160	.92					
		T	178	158	3.5	118	.66					
	800	L	168	151	1.5	110	.65	170	155	2.0	131	.77
		T	175	154	2.5	114	.65	179	158	2.5	102	.57
		L	166	150	2.5	127	.76					
		T	173	147	2.0	97	.56					
55	75	L	209	183	4.5	179	.86					
	650	L	190	170	1.5	132	.69					
65	-110	L	247	210	6.0	212	.86					
		T	264	227	7.5	164	.62					
	75	L	217	185	4.0	186	.86	244	224	2.0	180	.74
		T	232	187	5.0	115	.50	259	227	2.5	122	.47
	350	L	201	165	2.5	154	.77					
		T	215	177	3.0	107	.50					
	650	L	198	176	2.0	144	.73	207	188	2.0	118	.59
		T	213	186	2.0	88	.41	221	199	2.0	91	.41
	800	L	201	174	1.5	118	.59					
		T	213	185	1.5	69	.32					

(a) UTS, Ultimate Tensile Strength; Y.S., 0.2% Offset Yield Strength; N.S., Sharp-Notch Tensile Strength; Elongation in 2-inches.

(b) L, longitudinal; T, transverse.

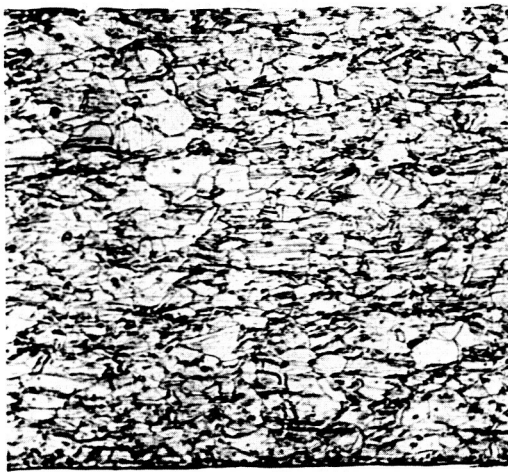


X100



X500

a) Cold reduced 40 percent - longitudinal



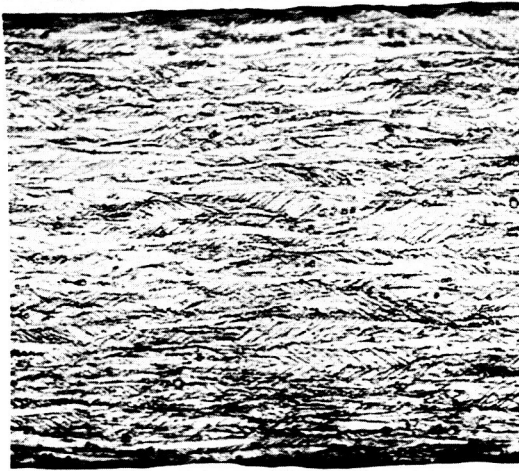
X100



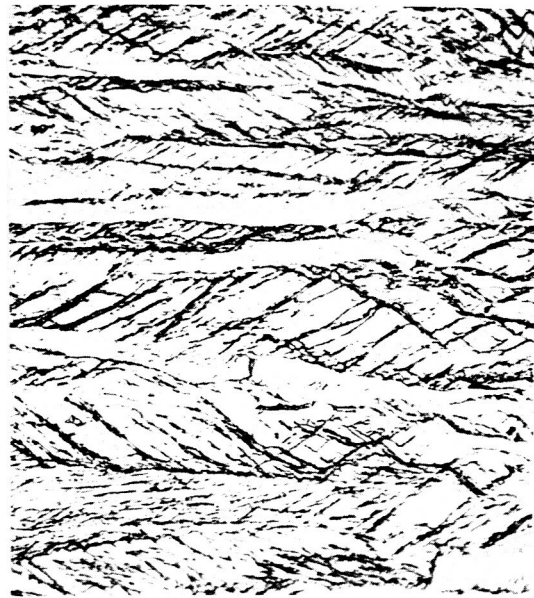
X500

b) Cold reduced 40 percent - transverse

Figure 1. - Microstructure of N155 alloy (Heat M-5623) cold reduced 40 percent and 65 percent. Material was aged 5 hours at 1200°F to delineate grain boundaries. Etched in 10-percent chromic acid.



X100



X500

c) Cold reduced 65 percent - longitudinal



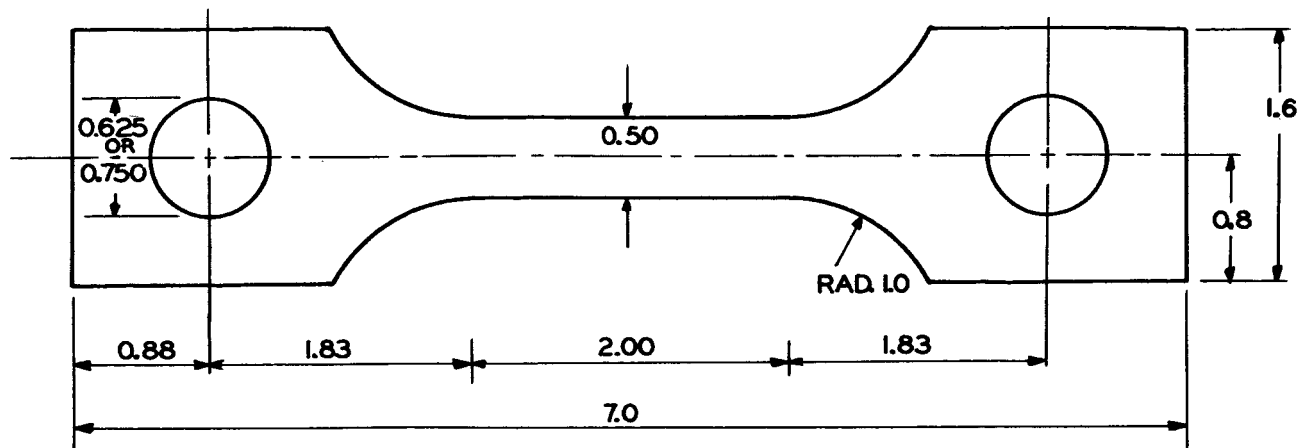
X100



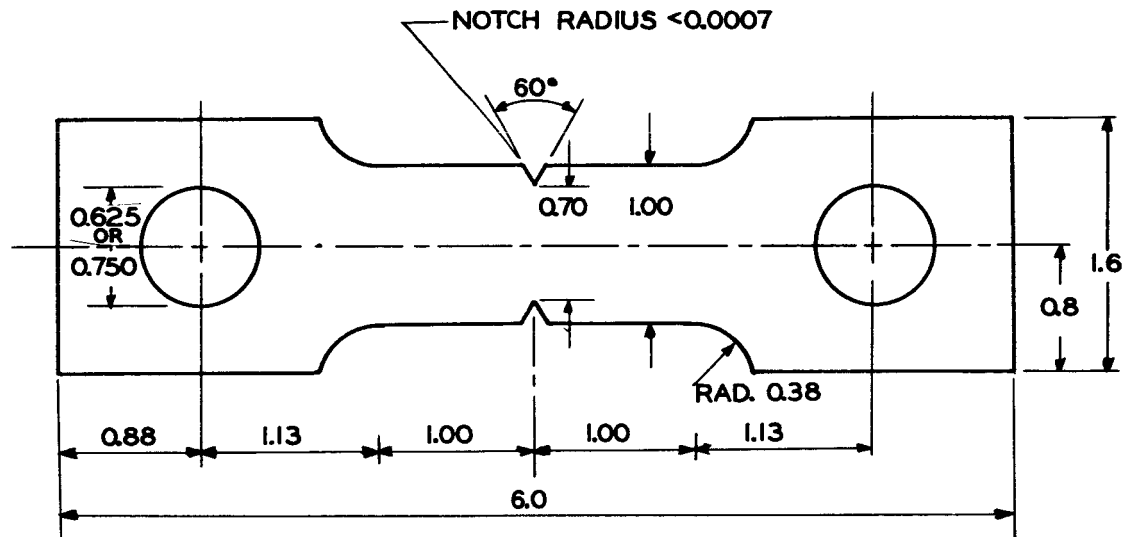
X500

d) Cold reduced 65 percent - transverse

Figure 1. - (Concluded) Microstructure of N155 alloy (Heat M-5623) cold reduced 40 percent and 65 percent. Material was aged 5 hours at 1200°F to delineate grain boundaries. Etched in 10-percent chromic acid.



a) Unnotched specimen



b) Specimen with sharp edge notches

Figure 2. - Test specimens used for screening tests. (All dimensions in inches)

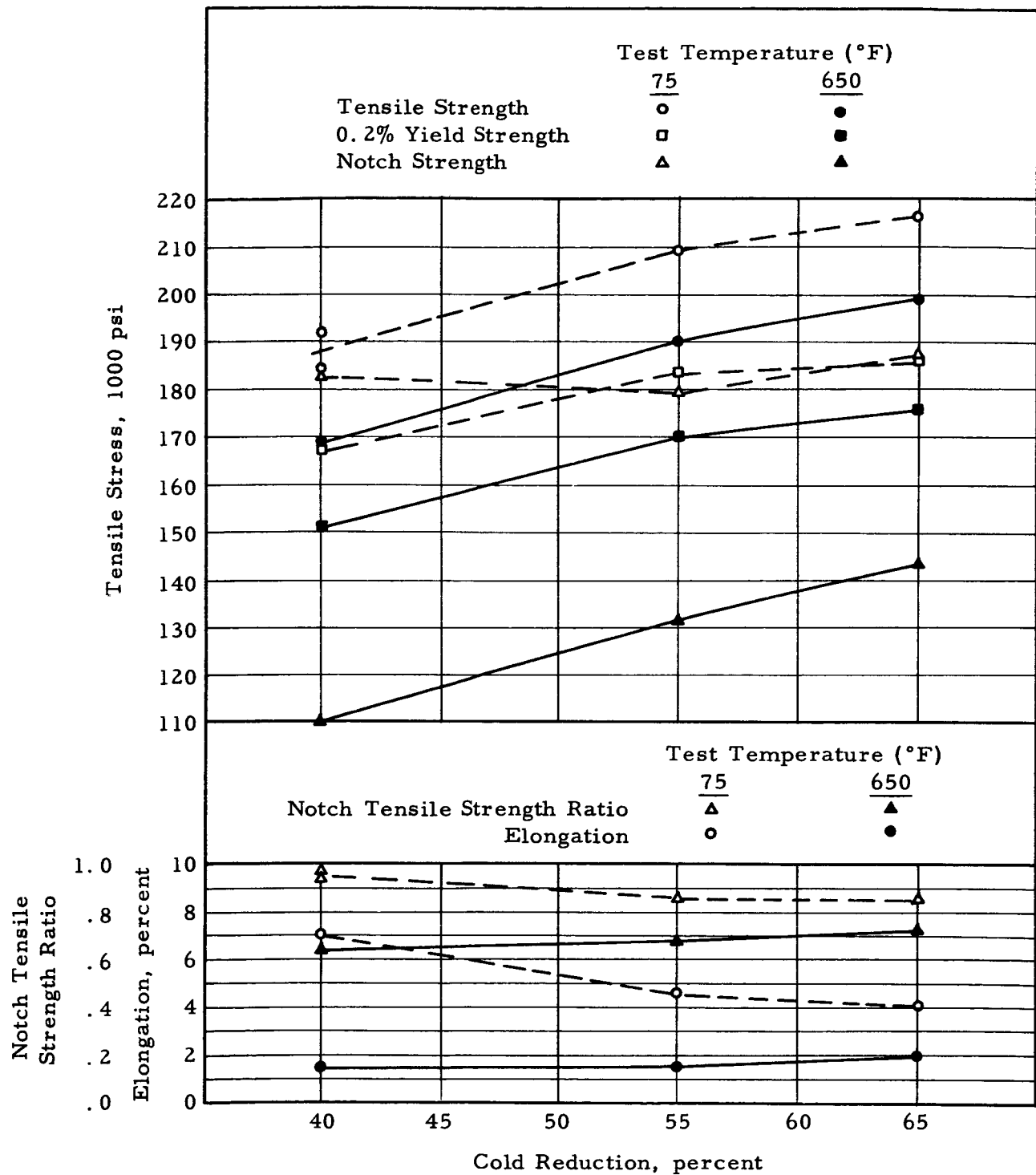


Figure 3. - Tensile properties at 75° and 650°F of N155 alloy (Heat M-5623) as a function of amount of cold reduction. (All test specimens from the longitudinal direction of 0.025-inch sheet)

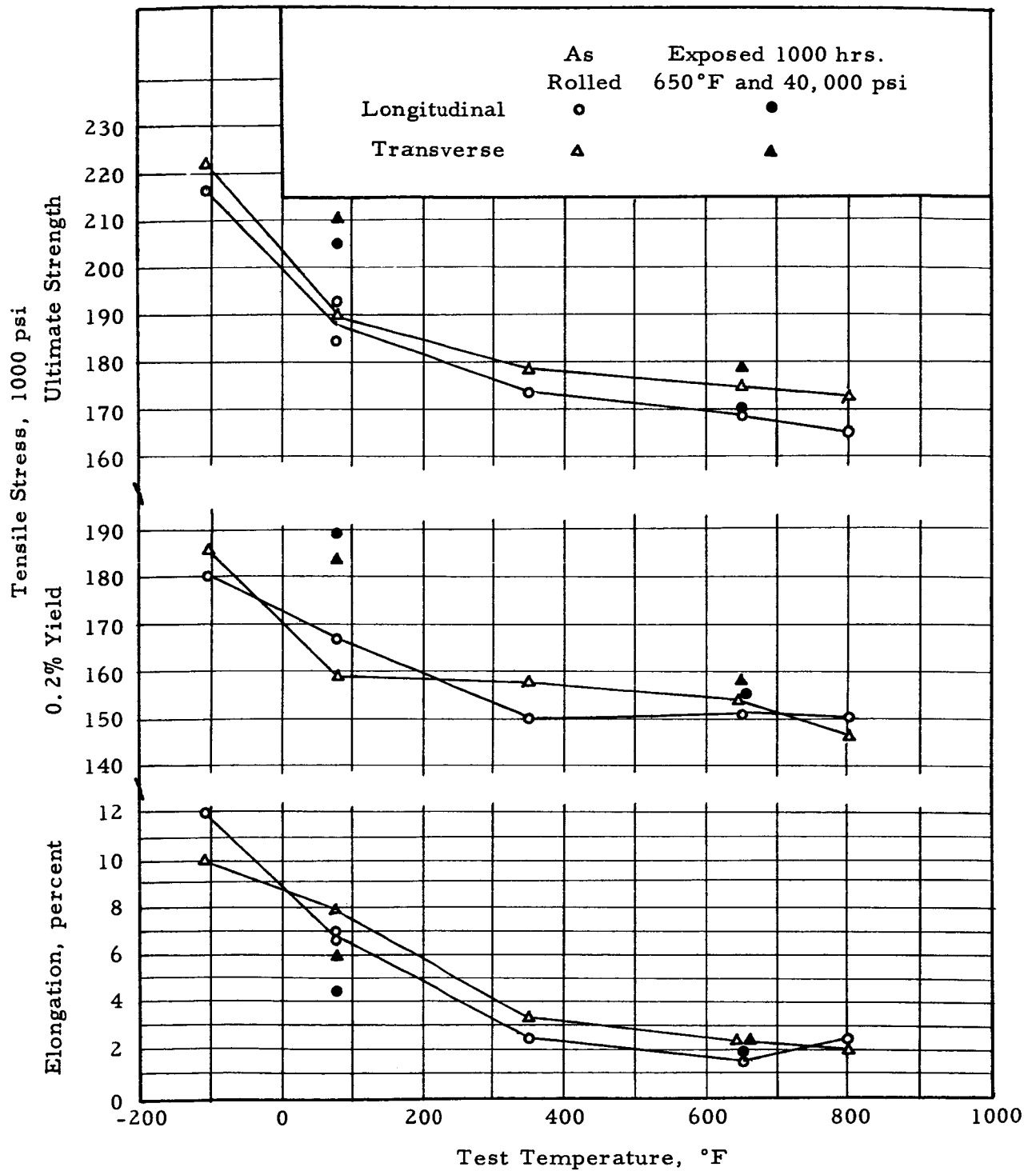


Figure 4. - Strength and elongation in tensile tests as a function of test temperature for unnotched specimens of N155 alloy sheet cold reduced 40 percent from Heat M-5623 in the as-rolled condition and after creep exposure.

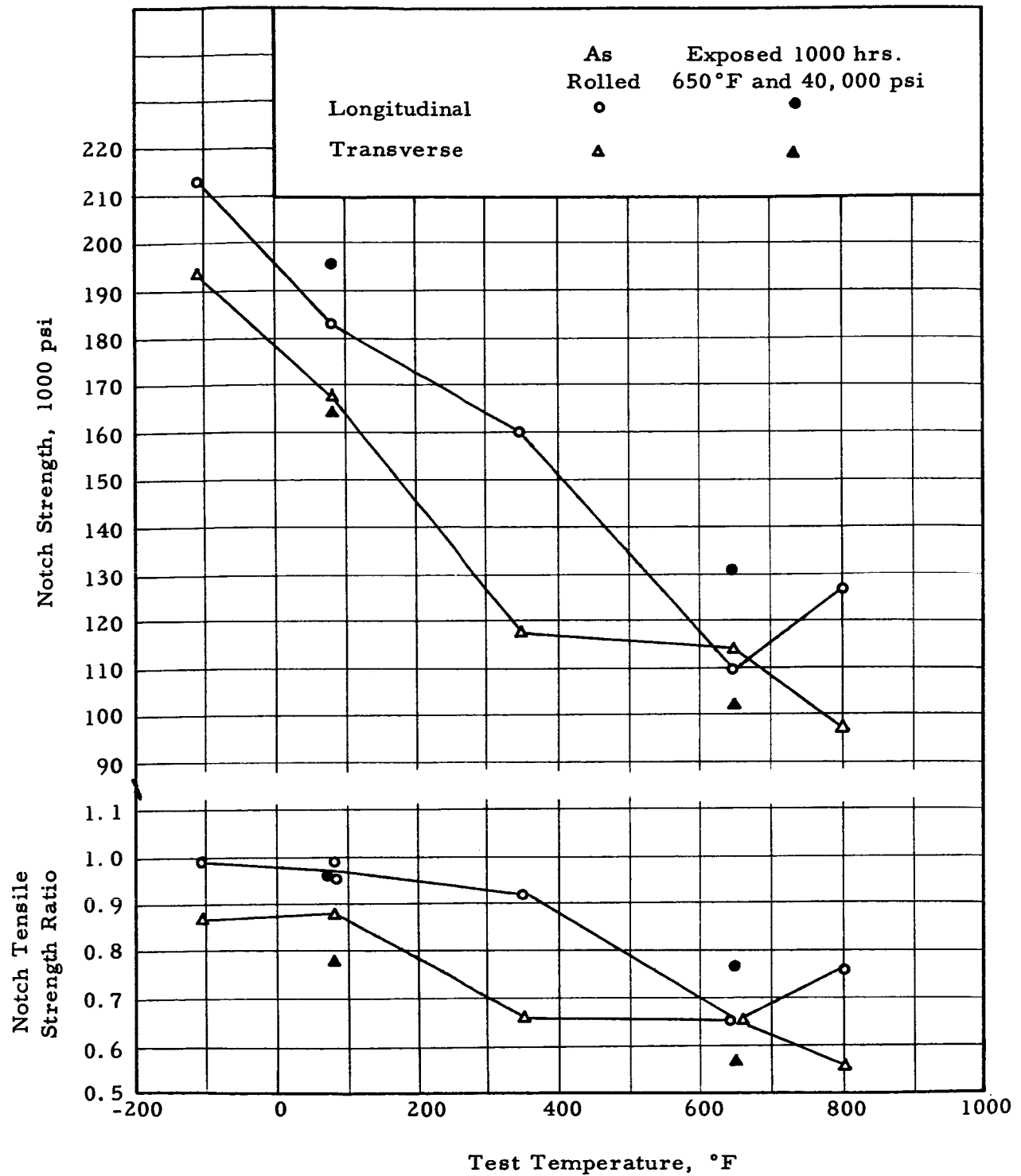


Figure 5. - Strength with sharp edge notches and notch tensile strength ratio as a function of test temperature for N155 alloy sheet cold reduced 40 percent from Heat M-5623 in the as-rolled condition and after creep exposure.



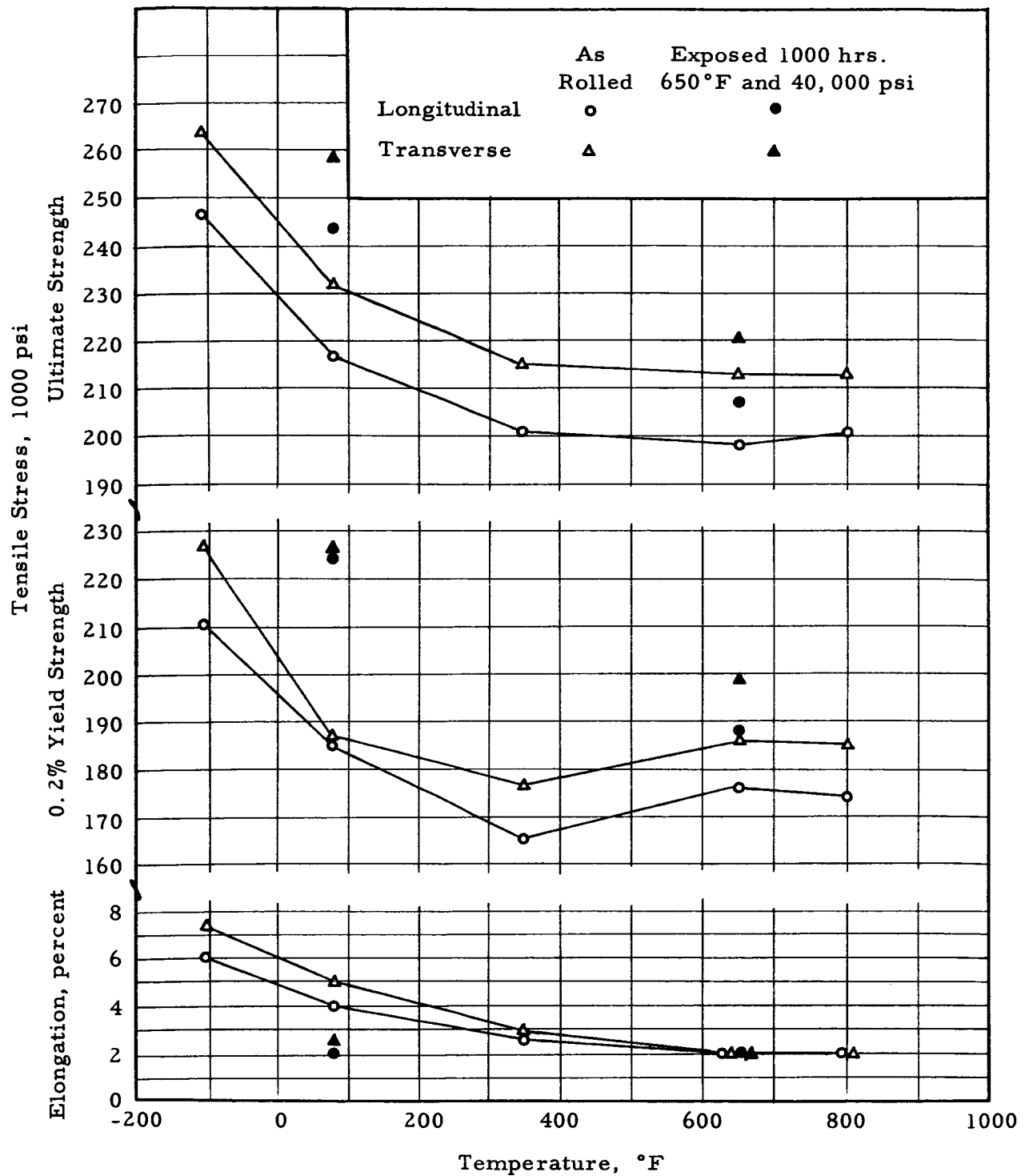


Figure 6. - Strength and elongation in tensile tests as a function of test temperature for unnotched specimens of N155 alloy sheet cold reduced 65 percent from Heat M-5623 in the as-rolled condition and after creep exposure.

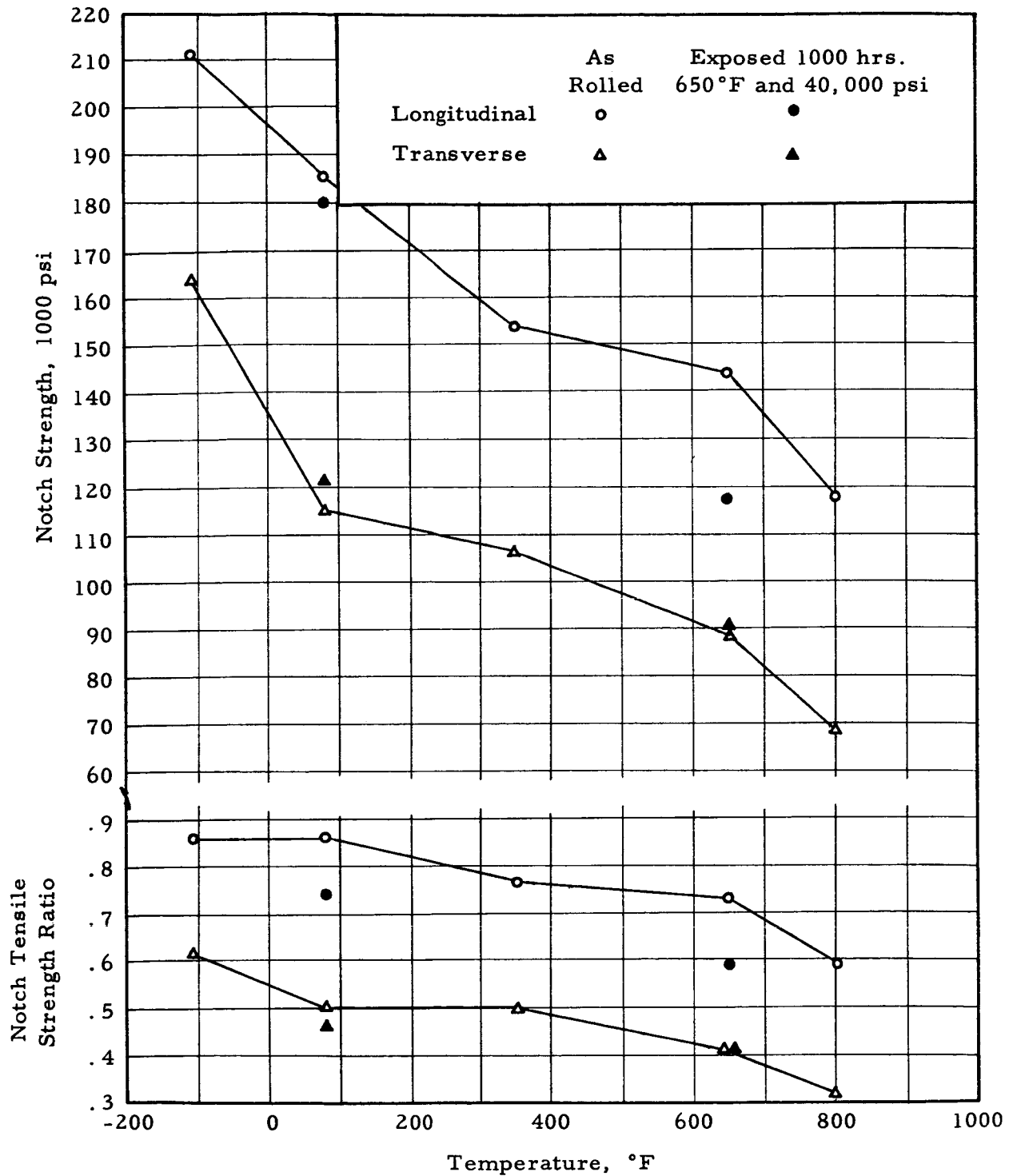


Figure 7. - Strength with sharp edge notches and notch tensile strength ratio as a function of test temperature for N155 alloy sheet cold reduced 65 percent from Heat M-5623 in the as-rolled condition and after creep exposure.

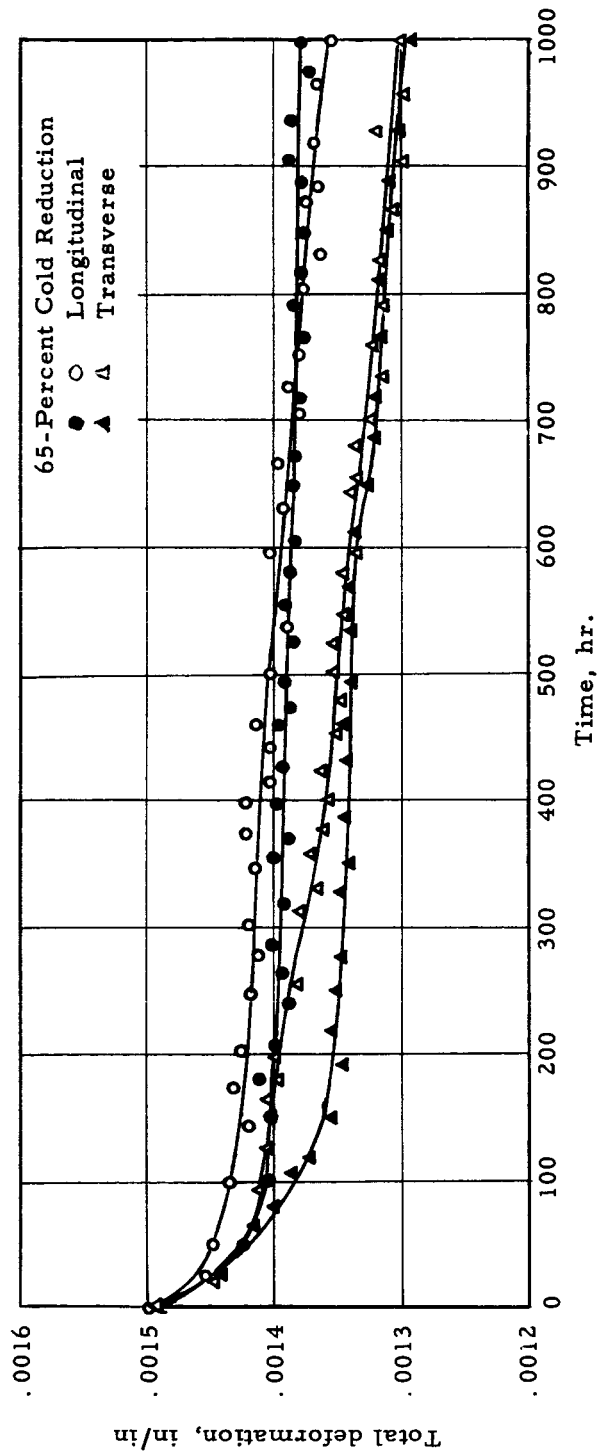
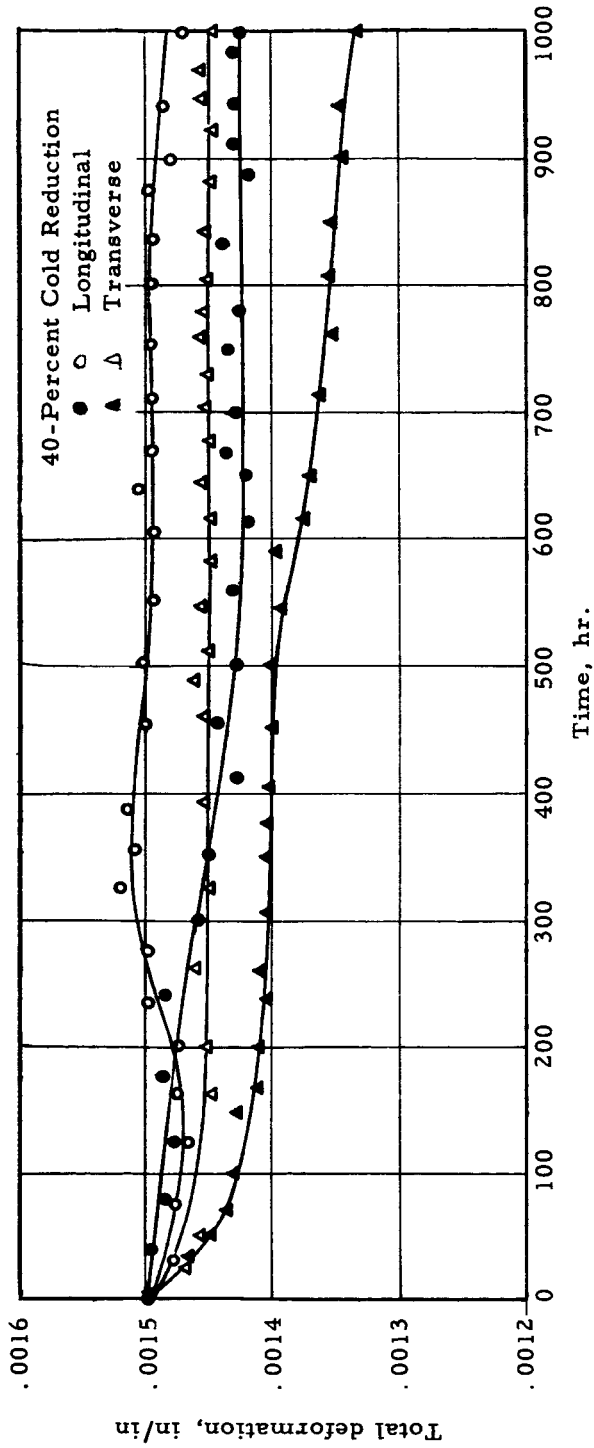


Figure 8. - Effect of time of exposure at 650°F under 40,000 psi on the total deformation of 0.025-inch thick cold-worked N155 sheet.

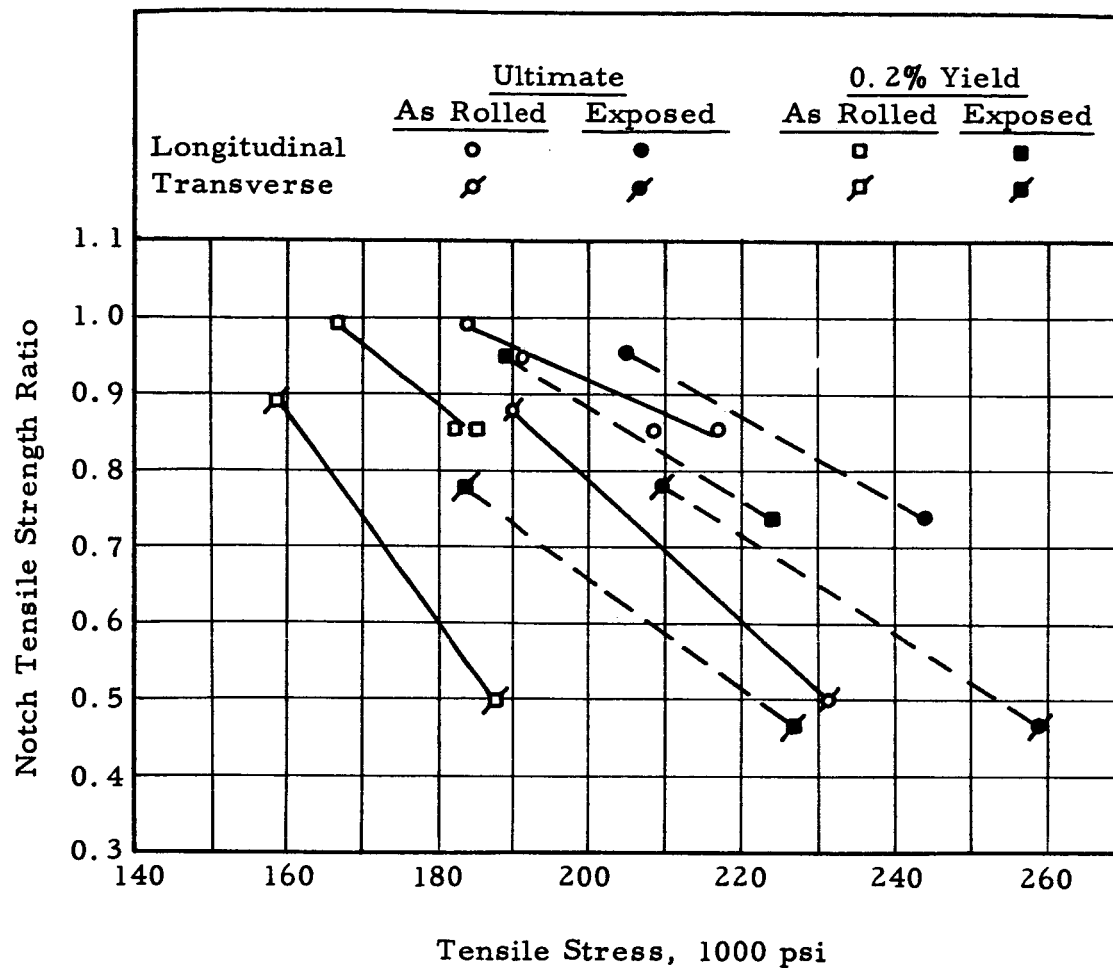


Figure 9. - Notch tensile strength ratio as a function of ultimate and yield strength at room temperature for N155 alloy in the as-rolled condition and after exposure for 1000 hours at 650°F under 40,000 psi.